Micro-Pak Enhanced Packaging Stickers: Enforceable Analytical Method

DATA REQUIREMENTS:

OPPTS 830.1800

AUTHOR:

John V. Simpson, Ph. D.

REVIEWED BY:

Eric W. Uffman, Ph. D.

STUDY COMPLETION DATE:

11/29/2012

PERFORMING LABORATORY:

Chemir Analytical Services

LABORATORY TEST IDENTIFICATION:

Sodium Metabisulfite by ICP-OES

DATA SUBMITTER:

Micro-Pak Ltd.

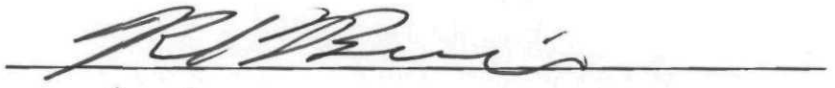
STATEMENT OF DATA CONFIDENTIALITY CLAIMS

No claim of confidentiality, on any basis whatsoever, is made for any information contained in this document. I acknowledge that information not designated as within the scope of FIFRA Section 10(d)(1)(A), (B), or (C) and which pertains to a registered pesticide is not entitled to confidential treatment and may be released to the public, subject to the provisions regarding disclosure to multinational entities under FIFRA 10(g).

Company: **Micro-Pak Ltd.**

Company Agent: Robert S. Brennis
Brennis Consulting Services LLC

Title: Study Submitter and Agent for
Micro-Pak Ltd.

Signature: 

Date: 12/2/12

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

This report only concerns the development of an enforceable analytical method for this product. There are no Good Laboratory Practice Standards (GLPS) specifically identified for the development of this data. Therefore, the Good Laboratory Practice Standards (GLPS), as specified in 40 CFR Part 160, are not applicable.

Author: _____ Date: 11/30/12

Name of Signer: John V. Simpson

Name of Company: Chemir Analytical Services

Company: Micro-Pak Ltd.
Company Agent: Robert S. Brennis
Brennis Consulting Services LLC

Title: Study Submitter and Study Sponsor
Agent for Micro-Pak Ltd.

Signature: _____

Date: 12/2/12

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An analytical method to verify the certified limits for the total amount of Sodium Metabisulfite in the Enhanced Packaging Stickers is presented below. This method uses a microwave enhanced digestion followed by inductively coupled plasma-optical emission spectroscopy (ICP-OES) to quantify the amount of sulfur present in Micro-Pak Enhanced Packaging Stickers. The sodium metabisulfite concentration is then calculated from this determined sulfur value.

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Quality Control Procedure

Standard Preparation and Calibration

At each test period, prior to sample analysis, a standard calibration curve is generated using 20, 100, and & 500 mg/L solutions. These standard solutions are prepared by volumetric dilutions from a 10000 mg/L sulfur stock solution (Inorganic Ventures Lot: E2-S01120) along with an addition of a 1000 mg/L scandium internal standard solution that results in a final Sc concentration of 1mg/L. A quality control standard is also prepared separately at a concentration of 100 mg/L from a 10000 mg/L sulfur solution procured from a second source (SCP Science Lot: S120116017).

Test Substance Preparation

Approximately 0.1 g of the sample is transferred to a microwave digestion tube. A digestion solution is then added to the tube consisting of 5 ml of HNO_3 (68-70%), 2 ml of H_3PO_4 (85.6%), 1 ml H_2O_2 (30%), and 5 ml of deionized water. The mixture is then microwaved according to the method outlined in Table 1 using an Anton Paar Multiwave 300 (SN:80839927) with a HF 100 rotor (SN:80122555).

Table 1: Microwave digestion program

Phase	*Temp. (ºC)*	*Ramp (min)*	*Hold (min)*	*Fan*
1	*200*	10	30	1
2	*0*	-	30	3

Once the digestion method is complete, the sample solution is then transferred to a 50 ml test tube, and 0.05 ml of a 1000 mg/L scandium internal standard is added. Then the solution is brought to volume with water.

Each sample is prepared in triplicate and analyzed in triplicate. From the determined sulfur concentration, the amount of sodium metabisulfite present in the same was calculated using the assumption that all sulfur present was in the form of sodium metabisulfite (see calculation below).

$$Metabisulfite\ Conc.\ in\ Sample\ (mass\%) = Sulfur\ Conc.\ (\%) \times \frac{Sodium\ metabisulfite\ mass\ (190.107\ g/mol)}{Sulfur\ mass\ \left(32.06 \frac{g}{mol}\right) x2\ sulfur}$$

Method Performance

Table 2 lists the wavelengths used during the analysis. The reported values are the result of the average of all wavelengths listed in Table 2 and have a maximum RSD of 3%.

Table 2: Spectroscopic Analysis Parameters

Element	*Wavelength (nm)*
S	*181.975, 180.669, 182.563*
Sc (as IS)	*424.683*

Linear regression of the calibration curve provides a correlation coefficient with a minimum value of 0.999. The quality control standard was run at the beginning and the end of the sample analysis with an acceptance criterion of ±10%. The reported values of the analysis of two samples are present in Table 3.

Table 3: Sodium Metabisulfite concentration replicate analysis

Sample A					*Sample B*				
Prep.	*Run*	*Metabisulfite Conc. (%)*	*Avg.*	*%RSD*	*Prep.*	*Run*	*Metabisulfite Conc. (%)*	*Avg.*	*%RSD*
1	*1*	19.57			1	*1*	20.16		
1	*2*	17.91			1	*2*	19.06		
1	*3*	17.40	18.29	6.19	1	*3*	19.03	19.42	3.31
2	*4*	18.83			2	*4*	20.34		
2	*5*	16.78			2	*5*	19.92		
2	*6*	18.71	18.11	6.34	2	*6*	19.09	19.79	3.20
3	*7*	18.95			3	*7*	20.04		
3	*8*	14.97			3	*8*	-		
3	*9*	18.50	17.47	12.46	3	*9*	19.63	19.83	1.58
Average		17.96			*Average*		19.66		
%RSD		7.823			*%RSD*		2.716		